



Agricultural Research Institute, Pusa

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# THE INDIAN SALTPETRE INDUSTRY

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# THE INDIAN SALTPETRE INDUSTRY,

## INTRODUCTORY.

The production of potassium nitrate, saltpetre, in India doubtless dates from very early times, but no accurate accounts of it exist. The industry has suffered some fluctuations. Fifty or sixty years ago the average export from Calcutta amounted to over 30,000 tons, but has usually averaged about 20,000 tons for a long period. There is some indication that the average outturn is slowly declining, though the decrease is not large. The price has remained very constant and shows no sign of falling. The following statement illustrates these points :—

Period.	Average annual export.	Average price per cwt.
	Tons.	R. a. p.
1894-98 . . . . .	20,400	11 0 0
1902-06 . . . . .	18,400	10 6 0
1907-10 . . . . .	18,400	11 4 0

The Indian production is probably only a small part of the total world's production, but that there is a real demand for potassium nitrate is established not only by the stability of the market price, but also by the fact that Germany manufactures considerable quantities by acting on Chili saltpetre with potassium chloride.

The Indian industry depends for its raw material on earth which is collected from old, as also from existing, village sites. Such earth is scraped together in small quantities by a very poor caste called "Nuniah" or "Lunia" who extract from it the saltpetre. No doubt the nitrates, which this earth contains, are formed from organic waste materials, urine, dung and vegetable debris, but our knowledge does not go further than this. No bacteriological investigation has been made regarding the

changes which occur in these earths. For instance we do not know why such earth is found only in certain localities. From such village earth the Nunia extracts a crude saltpetre; this is purchased by the refiner, who prepares from it the much purer grade for export.

It is the purpose of this Bulletin to give first a description of the methods employed by (a) the Nunia, and (b) the refiner, and to subsequently describe a new method and apparatus for refining the crude saltpetre, which has recently been devised in the Chemical Section of the Agricultural Research Institute at Pusa.

It will be a convenience if, before proceeding to a description of the industry, a few words are said about the term "salt." The term "salt" or "common salt" refers generally to a certain substance universally consumed both by man and animals with their food. But if a chemist examines such materials as saltpetre earth, he will say that they contain not one salt but several "salts" of which "common salt" is only one. There is no necessity to enter here upon any explanations as to what sort of substances he describes as "salts." The case will be sufficiently met if it is stated that practically all the various substances that we shall have to refer to in this Bulletin are termed by him "salts". Each of these has its special name and characteristics. The most important to us on the present occasion are the following :—

1. Potassium nitrate, or "Indian Saltpetre."
2. Sodium nitrate or "Chili saltpetre."
3. Calcium nitrate.
4. Sodium chloride or "Common salt."
5. Sodium sulphate or "glauber's salts."

Another matter which is all important to a correct understanding of the saltpetre industry are the terms "soluble," "solubility," "saturated solution." The Hindustani verb "gul jana" means "to dissolve" and the phenomenon of solution is well known to the refiners. If a substance is added to water and is found to disappear in the water, it is said "to dissolve." All the salts above referred to are "soluble" in water. But when we go into the subject more deeply, and enquire how much of each will dissolve in a certain amount of water, we meet with differences which are of great importance to the processes which we shall describe.

Consider first the salt, potassium nitrate. If it is added in small portions to 100 parts of good well-water, it will continue to dissolve

until a certain amount has been added, when the water will fail to dissolve any more. Well-water in India has commonly a temperature of about  $25^{\circ}\text{C}$ . At this temperature the 100 parts of water would dissolve about 37 parts of the nitrate. Such a solution is called a "saturated" solution at  $25^{\circ}\text{C}$ . If the water is warmer, say  $32^{\circ}\text{C}$ , the 100 parts will dissolve about 48 parts of nitrate. This is a "saturated" solution at  $32^{\circ}\text{C}$ . If the water is heated to the temperature  $100^{\circ}\text{C}$ , it will take up no less than 247 parts of the nitrate. This is a "saturated solution" at  $100^{\circ}\text{C}$ . This example will serve two purposes. The term "saturated" solution is defined. Also an illustration is given of the effect of temperature on the amount of a salt which will dissolve in water.

Turning to the other salts that we are interested in, the solubility of potassium chloride is as follows; 100 parts of water will dissolve about 36 parts of this salt at  $25^{\circ}\text{C}$ , 38 parts at  $32^{\circ}\text{C}$  and 56 parts at  $100^{\circ}\text{C}$ . In this case the 100 parts of water at  $25^{\circ}\text{C}$  dissolve about as much potassium chloride as of potassium nitrate. The hot water dissolves more of the chloride than the cold water does, so that we have another example of the effect of heat; but the hot water only dissolves about one-fifth as much of the chloride as of the nitrate. It is a very common property of substances that they are more soluble in hot water than in cold.

In the case of common salt, the effect of temperature is only slight; 100 parts of water dissolve 36 parts of salt at  $25^{\circ}\text{C}$ , 37 parts at  $32^{\circ}\text{C}$  and 40 parts at  $100^{\circ}\text{C}$ .

In some cases there is a *decreased* solubility with a rise of temperature. Sodium sulphate is an example; 100 parts of water at  $25^{\circ}\text{C}$  dissolve 28 parts of the salt, at  $33^{\circ}\text{C}$  about 50 parts and at  $100^{\circ}\text{C}$  only 10 parts. Thus, this salt possesses the peculiar property of being more soluble at  $33^{\circ}\text{C}$  than in either warmer or cooler water.

Again, sometimes the presence of one salt enables the water to dissolve more of a second salt than it would do if the first salt were absent. Thus boiling hot water containing sodium chloride will dissolve one-fifth more potassium nitrate than it can do in the absence of the sodium chloride. Conversely the solubility of potassium chloride is *decreased* in the presence of potassium nitrate, and sodium sulphate is much less soluble in water containing sodium chloride than in pure water. These solubility relations, as will become apparent, are of the utmost importance to the saltpetre industry.

## PART I.

*The Production of Crude Saltpetre.*

The earths from which the crude saltpetre is obtained, contain very varying amounts of nitrate. Mr. Hooper\* quotes the analyses of a large number of samples in which the nitrate varied from as little as 1.0 per cent. up to as much as 29 per cent.; but the general run of these earths does not contain much more than 3 to 5 per cent. In addition to the nitrate, the earth always contains several per cent. of chloride and sulphate.

The process employed by the Nunia in the extraction of the nitrate is as efficient as it is interesting.

In descriptions of the method the terms "maceration," "lixiviation" have been used† but these are very misleading. If the earth were "mixed" or "macerated" with water, the quantity of water employed would certainly approximate to that of the earth, and hence the solution obtained would contain only some 5 per cent. or so of nitrate and frequently less. Moreover the earth could not be efficiently separated from the solution without much more costly appliances than these people could afford.

The following is a brief description of the Nunia's method :—

He builds up an earthen chamber called the "*Kuria*" or "*Kolhi*" with wet mud which is allowed to dry. This chamber has either circular walls some 5 or 6 ft. diameter, or oblong walls, and a floor which slopes somewhat from back to front. In the front wall is a hole at the level of the bed which allows the nitrate liquor to drain away (see plates I and II). Above the bottom of this earthen chamber is laid, resting on a few loose bricks, a "false bottom" of bamboos and matting. Occasionally I have seen some grass employed, but this does not lead to efficiency and is probably only employed to fill holes in damaged matting.

On this matting is then laid the saltpetre earth, and over this is poured water. Now the efficiency of the process depends on the mode of filling the earth into the "*Kuria*." If it were imperfectly put in,

\* Agricultural Ledger, 1905, page 23.

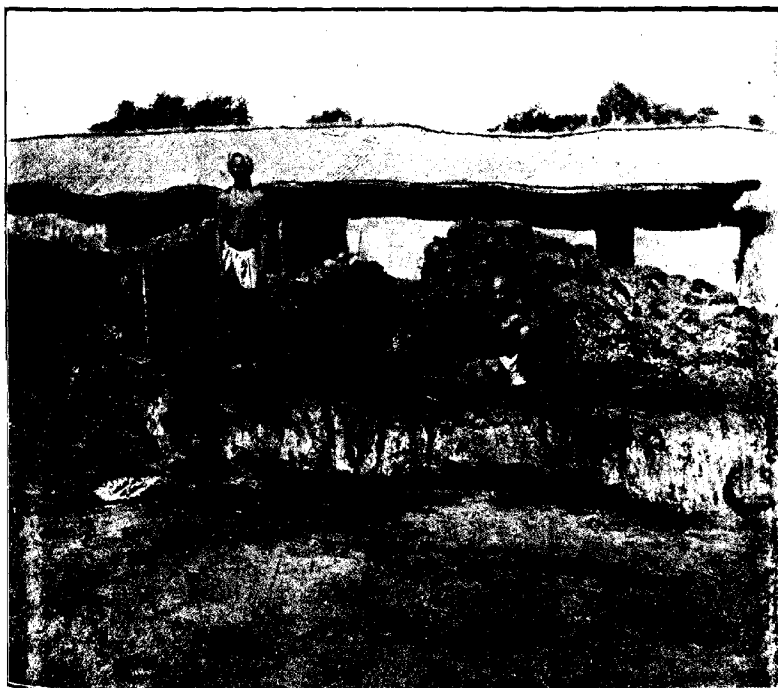
† Dictionary of applied chemistry, Vol. III, page 20.

PLATE I.



KOTHIA PERCOLATORS.  
Row of "Kurrias" or "Kothis."

PLATE II.







the water would create cracks or fissures and simply muddy water would run away below. The earth is filled in with the greatest care. It is *slightly* moist and is freed as far as possible from stones or fragments of bricks or pottery. First a portion, say a fourth part of the earth, is spread carefully over the matting, then the Nunia treads it most carefully all over (see plate I!); more earth is spread over the first portion, followed by careful treading, and the process continued until a layer of some 6" or 8" has been introduced. Very generally some of the wood ashes from the fire place are mixed with the earth before filling. These earths often contain calcium and magnesium nitrate as well as potassium nitrate; the wood ashes contain potassium carbonate which would change any calcium or magnesium nitrate into the potassium nitrate. Water is now poured over the surface and, in order to avoid disturbing the earth, a small piece of matting is placed temporarily on the latter. The addition of water is continued until about 1" lies on the surface of the earth. The first added water soaks in fairly quickly, but the absorption soon becomes slower and subsequent additions are made at intervals.

The water soaks downwards through the earth and finally, after the lapse of several hours, reaches the matting from which it flows to the bottom of the "*Karia*" and out at the hole in the side. As it soaks through the earth, the soluble salts, the nitrates, chlorides, sulphates, dissolve in it and are carried downwards. The water which drains downwards first, takes up the major portion of the nitrate and chloride as it goes along, and consequently the water which *first* reaches the *under* surface of the earth contains a much higher proportion of nitrate than would have been the case if the earth had been "mixed" or "macerated" with the water. The first "liquor" to run away from the "*Karia*" is often quite a concentrated solution of salts. It is usually clear, for since it percolates very slowly, the earth is not disturbed. It is coloured brown because, in addition to the salts, it also dissolves brown organic matters.

Thus whereas any macerating process would produce a very weak solution, the percolating process which is employed produces a strong one. The former would require many times as much fuel to boil down the extract as is actually required for the strong solutions obtained.

So far we have only considered the *first* liquor which drains away from the "*Karia*." It is only this portion that the Nunia puts into his pan. As the water continues to percolate through the earth, there is less and less nitrate for it to pick up and consequently the liquor running away

becomes weaker. If the process were continued by addition of water at the surface, all the nitrate and other salts in the earth would be dissolved and would pass out of the "*Kuria*." But it only pays the Nunia to boil down strong solutions; he cannot afford the fuel which would be required to boil down the weaker ones. For this reason he only pours sufficient water on the surface of the earth in the "*Kuria*" to yield strong liquors. It follows that when the process stops he has (a) some strong liquor which he can afford to boil down, (b) usually some weaker liquor, (c) the wet earth in the "*Kuria*." It is evident that although (a) will often contain a large part of the nitrate which was in the earth when packed into the "*Kuria*," both (b) the weaker liquor and (c) the wet earth in the "*Kuria*" will contain some material portions. In order to avoid losing this nitrate, the weak liquor (b) is thrown on to the earth of the "*factory*," and the wet earth (c) is dug out of the "*Kuria*" and allowed to dry gradually. Eventually all this earth about the factory is broken up, air-dried and again put into the "*Kuria*" for extraction. In fact the process seems to be a never-ending one. The whole of the earth about the factory seems to consist of earth which has been already partly extracted and on to which small amounts of weak liquors are thrown.

Is it then the case that nitrification is constantly going on in these factory earths? If it were not so, the earths must become each time they are extracted in the "*Kuria*," weaker in nitrate. The Nunia always brings into his "*factory*" some fresh village earth, which is commonly or frequently mixed with old earth when packed into the "*Kuria*," which fact would account for some additions of nitrate. There is in fact at every factory a constant addition of earth, and a constant abstraction of nitrate. But it seems probable that, unless the whole were also aided in some way by nitrification at the *factory*, the earths would become weaker and weaker as time goes on. On the other hand no special additions of organic materials are made by the Nunia, and nitrification merely implies the change of organic nitrogenous compounds into nitrites and nitrates. One explanation would be that assimilation of atmospheric nitrogen goes on, coupled with nitrification, but that these processes are so limited in intensity that outside additions of nitre earth are also necessary. These are mere speculations, made only because, whilst it is easy to say that this industry depends on the nitrification of organic compounds, the facts surrounding the industry are not in reality explained so simply. It is a case which deserves a careful bacteriological study.

Returning to the strong liquor which first runs away from the "*Kerida*," this is boiled down in either an iron pan or an earthen one, or it is allowed, as in the Punjab, to concentrate in a shallow masonry "tray" in the sun.

As these liquors concentrate, they first deposit common salt, especially when the operation is conducted over a fire. In this case when sufficiently concentrated, the whole is allowed to cool and the saltpetre crystallises out. The mixture of common salt and saltpetre is separated from the mother liquor and sold as crude saltpetre to the refiner. The mother liquor is thrown on the factory earth and allowed to dry up. Although common salt, (sodium chloride,) becomes precipitated in this process, it is not separated by the *Nunia*, firstly because the Salt Department does not permit him to do it, and secondly because it would not pay him to separate it if he were otherwise free to do so.

The composition of the crude saltpetre naturally varies a good deal and this is illustrated by the following analyses.\*

	Farukhabad.		Okara.		Mozaffer- pore.	Burban- pura.
	I	III	I	III		
	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.
Potassium nitrate . . .	66.07	44.92	55.00	26.86	49.26	68.10
Calcium nitrate . . .	..	..	2.60	..	3.28	2.60
Magnesium nitrate . . .	2.54	4.80	..	12.24	7.44	2.12
Sodium chloride . . .	21.81	35.38	31.22	34.80	16.82	17.98
Sodium sulphate . . .	3.65	10.60	5.88	11.26	14.60	3.40
Insoluble matter . . .	.90	1.20	1.10	1.40	1.50	1.70
Water . . .	5.00	3.70	5.20	13.50	7.00	3.80
	100.00	100.00	100.00	100.00	100.00	100.00

\* Agricultural Ledger, Vol. XII, page 31.

## PART II.

### *The Production of Refined Saltpetre.*

The crude saltpetre produced by the Nunia is, I believe, all sold to the refiner. The refining processes are several in number, but the principal ones may be sketched thus. The crude saltpetre is added to a boiling hot "mother" liquor from a previous operation. Now such a liquor is saturated with common salt and sodium sulphate, but is able to dissolve large amounts of potassium nitrate and lesser quantities of potassium chloride, which latter salt, though not shown in the analyses of crude saltpetre detailed in the previous paragraph, is nevertheless frequently present.

Consequently when the crude saltpetre is added to the hot mother liquor, the potassium nitrate and other nitrates which may be present, and a small amount of potassium chloride are dissolved, but the sodium chloride and sulphate and the dirt remain undissolved. The fire is removed, and the insoluble matters quickly subside. The hot clear liquid is run into wooden vats where it is allowed to cool, and as it cools a good deal of the potassium nitrate crystallises out. The insoluble substances remaining in the pan are called "*sitta*," and are dealt with separately. After the liquor in the vats has cooled, the crystallised potassium nitrate—refined saltpetre—is taken out, drained from the liquor, often washed slightly with water and is ready for the market. The mother liquor is then used again for dissolving the nitrate from a second lot of crude saltpetre.

But whilst this is a rough sketch of the process, it does not include all the operations which are adopted, nor does it explain much that is of interest. For instance in practice it is found that the mother liquor cannot be employed over and over again, but only a limited number of times; common salt is thrown out at one stage when evaporating mother liquors; and again the "*sitta*," which contains much nitrate, has to be dealt with. In describing in greater detail the whole of the operations, it will be better to turn to the *nitre earth*, a large mound of which exists in every refinery. This mound of nitre earth originates with the "*sitta*," that is the portion of the crude saltpetre which, it has been mentioned, is insoluble in the hot liquor. It consists very largely of salts, sodium sulphate and chloride and potassium nitrate, besides common

earth. It has been generally assumed that saltpetre "grows" in this earth, but for the moment this question need not be discussed. It is merely necessary to remember that, with present methods, this earth is a necessary adjunct to every refinery. From this nitre earth, the nitrate is extracted in precisely the same manner as that adopted by the Nunia, that is, it is packed with great care into "*Kurias*;" usually some wood ashes are mixed with this earth, for the reason which has already been given (page 5); and by allowing cold water to percolate through it, a part of its nitrate is extracted as a strong liquor, which is boiled down. In addition there is some weak liquor produced and there is also the wet earth to be dug out of the "*Kuria*" again, containing likewise some of the nitrate. The "*Kurias*" in refineries are better made than those of the Nunia and they are somewhat larger, but in other respects they are similar.

The following analyses illustrate the composition of the strong liquor first running away from the "*Kurias*":—

	Pusa.	Barhan- pura.	Savan.	Bukraman.
	Per cent.	Per cent.	Per cent.	Per cent.
Potassium nitrate . . . .	15.21	24.82	15.33	7.17
Potassium chloride . . . .	3.69	.46	2.21	2.61
Sodium chloride . . . . .	15.46	25.89	15.37	19.89
Calcium chloride . . . . .	...	...	.33	.89
Magnesium chloride . . . .	1.64	1.99	1.84	...
Calcium sulphate . . . . .	.88	.41	.75	...
Magnesium sulphate . . . .	.17	.51	...	.59

The first strong liquor from the refinery "*Kuria*" is dealt with in two different ways. In one of these it is boiled in iron pans, and as the water evaporates common salt is precipitated. This salt is taken out, and the hot and strong solution containing the nitrate allowed to cool in

wooden vats or earthen jars, when potassium nitrate crystallises out. This refined saltpetre is called "*kuthia shora*" and is apparently not very pure, as the following analyses illustrate :—

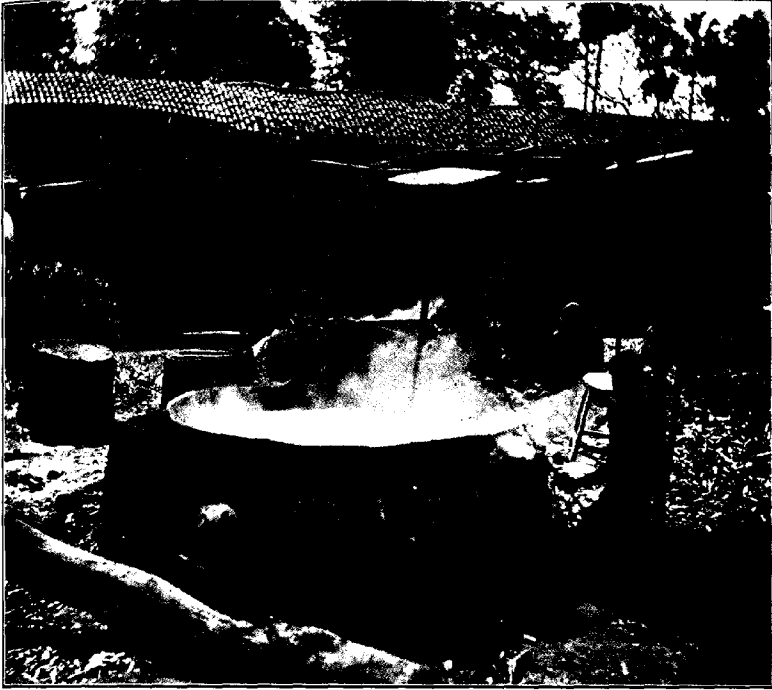
	Burhanpura.	Savan.
	Per cent.	Per cent.
Potassium nitrate . . . . .	65.21	50.95
Sodium nitrate . . . . .	7.74	—
Potassium sulphate . . . . .	—	1.68
Sodium sulphate . . . . .	.61	—
Potassium chloride . . . . .	—	19.81
Sodium chloride . . . . .	22.92	20.45

In other refineries, as in the "*Gundi Galai*" process, the strong liquor from the "*kurias*" or "*kothis*" is put into a pan some 16 ft. diameter and with it is heated the "*sitta*" of a previous operation. The hot liquor dissolves from the "*sitta*" the nitrate it contains and also a good deal of the common salt, etc. The clear hot liquid is separated from the mud and the latter then passes to the heap of refinery earth, for however carefully the hot liquid is separated from the mud, the latter must always carry some liquor with it and hence some nitrate.

The cleared liquid is boiled down. (See plates III and IV). This hot liquid is far from being saturated with nitrate, in fact three-fourths or more of the water might be evaporated off before any saltpetre would crystallise out at the boiling temperature. With respect to the sodium chloride however the case is different, for it is nearly or quite saturated with this salt, and consequently as the water is boiled off, the common salt becomes precipitated. This is then "*fished*" out with ladles as in the first process named.

To the hot liquor now obtained is added some mother liquor from the crystallising vats, and to this mixture, which at the high temperature is able to dissolve large amounts of nitrate, is added crude saltpetre. The

PLATE III.



EVAPORATING LIQUOR FROM THE KOTHIA.

PLATE IV.









PLATE V.



Photo. Engraved & printed at the Offices of the Survey of India, Calcutta, 1911.

SALTPETRE CRYSTALLISING IN VATS.

mixture is boiled for a few minutes and then the fire is removed from below the pan. The insoluble matters, consisting mostly of common salt and earth, are allowed to settle, and the clear brown and hot liquid is run into the wooden vats already mentioned.

These vats are usually rectangular, less often square, and measure some 7 ft.  $\times$  5 ft., or 6 ft.  $\times$  6 ft., and about 2 ft. deep. Sometimes also they are small wooden tubs as in plate V. As the liquid cools in these vats, the dissolved salts crystallise out. Chief among these is, of course, the potassium nitrate; to a much less degree will sodium chloride and potassium chloride crystallise. It is of importance from the refiner's point of view to allow the liquid to crystallise *slowly*. He has no appliance for separating the crystals from the mother liquor very efficiently and can only drain the liquor away. If he produced small crystals these would, on account of their larger total surface, retain more liquor than if large crystals are produced. In order to produce large crystals, the hot liquor must cool slowly. To this end he frequently embeds the wooden vats in earth. He sometimes employs another device which is very interesting. If the hot liquor were simply exposed to the air, evaporation, which of course must always take place, would be considerable. Now assuming that *no* evaporation were allowed, and since sodium chloride (which is the chief impurity) is nearly as soluble in cool as in hot water, this salt would only separate with the nitrate to a limited extent. If however the water were allowed to evaporate, its sodium chloride would separate and increase the contamination with the saltpetre. In order to prevent in a measure evaporation from the surface of the cooling liquor, the refiner pours carefully on to the surface of the hot concentrated liquor in the vat, some cold water. This water is lighter than the nitrate liquor and therefore floats on the surface, and the evaporation which takes place is provided for by this added water. To a certain extent this water must naturally get mixed with the nitrate liquor, but added dexterously as is done by these men, the water remains fairly distinct on the surface. Whether water is added in this manner or not, the top liquid soon forms a protection to that below, for it cools first, deposits its nitrate and is then specifically lighter than the warmer liquid underneath; the upper layer then forms a non-conducting shield to the lower liquid.

The crystallisation process proceeds generally for some 8 or 10 days before it has cooled to about 35°C or so when the refined saltpetre is

separated. This refined saltpetre, called "*Kalmi shora*," is usually light brown coloured and fairly pure as the following analyses exemplify :—

	I.	II.	III.	IV.
	Per cent.	Per cent.	Per cent.	Per cent.
Potassium nitrate . . . .	82.61	96.17	91.19	92.98
Potassium chloride . . . .	11.01	...	3.54	1.87
Sodium sulphate . . . . .	...	.15	...	.61
Sodium chloride . . . . .	3.61	.86	.61	3.33

*Washing saltpetre. The "dholai" process.*—This "*Kalmi*" saltpetre is, in some refineries, subjected to a farther purification by washing. For this purpose it is placed in sacks which rest over tubs or over a tank (see plates VI and VII) and the sack mouth having been opened, a weak liquor from the tank is poured over it. This weak liquor probably serves two purposes; it washes the saltpetre partly and it also wets the whole sack full of crystals thoroughly. This preliminary treatment is continued for an hour or more, the man passing from one to another of a line of such sacks. Thereafter a small quantity of cold water (in one case I found it to be about one gallon) is poured dexterously over the saltpetre at the top of the sack and allowed to drain through. Naturally this process dissolves some of the nitrate as well as some of the sodium chloride, the removal of which is the principal object of the washing, but the result is the production of a purer saltpetre. The following analyses exemplify the effect of the process :—

	BURIJANPURA.		SAYAN.		BAKRAMAU.	
	Un-washed.	Washed.	Un-washed.	Washed.	Un-washed.	Washed.
Potassium nitrate . . . .	90.70	94.91	81.98	91.55	88.63	94.70
Potassium sulphate . . . .	...	...	5.44	.93	.15	.15
Sodium sulphate . . . . .	.91	.08	...	...	..	...
Potassium chloride . . . .	...	...	2.59	2.51	6.06	2.67
Sodium chloride . . . . .	5.40	3.12	7.05	1.68	.07	.10
Sand . . . . .	.80	.20	.26	.35	...	...

PLATE VI.



WASHING SALTPETRE IN SACKS, MUZAFFARPUR, 1906.

PLATE VII.



Photo. Engraved & printed at the Offices of the Survey of India, Calcutta 1911.  
WASHING SALTPETRE IN SACKS, MUZAFFARPUR, 1906.



The *bye-products* of the refinery have been incidentally mentioned, but deserve a more collected reference. The substance "*sitta*" has been described. The following analyses illustrate its composition :—

	Muttra.	Farrakha- bad.	Farrakha- bad.	Patna.
	Per cent.	Per cent.	Per cent.	Per cent.
Potassium nitrate . . . .	14.0	4.84	6.70	8.20
Potassium sulphate . . . .	3.68	5.96	6.90	21.10
Potassium chloride . . . .	22.57	...	26.11	...
Sodium sulphate . . . . .	...	5.61	...	5.14
Sodium chloride . . . . .	35.80	74.33	46.75	44.52
Water . . . . .	15.72	5.98	9.86	6.31
Organic matter . . . . .	.18	.21	.21	.70
Sand . . . . .	5.29	.95	2.04	13.16

It has been explained that this "*sitta*" is either mixed with the refinery earth or treated with liquors from the "*Kothis*" when much of the salts in it are dissolved. In the latter case however these, more particularly the common salt, are separated subsequently.

*Common salt* is a second bye-product of the refinery. This like the "*sitta*" is not a voluntary product. As refining is carried out to-day the refiner cannot help separating the sodium chloride and a little consideration will show that it must not only be taken out but it must also be purified. As a matter of fact after fishing it out as described, on pages 9 and 10 it is rapidly washed with cold water. I estimated that about one-third of the salt is thus redissolved. The question remains then why is it purified? As taken out of the pan of hot liquor, each one hundred parts of the salt cannot retain with it less than about 25 parts of liquor. This liquor is a very concentrated solution of potassium nitrate. The amount of nitrate in it will vary, but there will not be less than some 15 to 20 parts of potassium nitrate in it. Which then is the more valuable, the 100 parts of salt or the 15 to 20 parts of potassium nitrate? A maund of good salt is worth, apart from Government duty, about 4 annas; 13.16 lbs. of potassium nitrate, which is about the equivalent of the



above proportions, is worth about R1 to R1-4-0. There is no doubt that the real reason for washing and purifying the common salt of the refinery is to get the nitrate away from it rather than to produce good salt.

The third bye-product of the refinery is the waste liquor. There are waste liquors from the "*Kuria*" and waste mother liquors. The former are so dilute that it pays best to throw them on to the refinery earth and let them dry up in the sun. The mother liquors from the crystallising vats are used once or twice over again for dissolving crude saltpetre, but thereafter they are considered to become too impure and are likewise thrown on to the refinery earth.

Thus in the refinery there are constant additions of waste products, all containing considerable amounts of nitrate, to the heap of refinery earth. In addition, owing largely to imperfect appliances, nitrate gets spilled about on every side, with the result that not only in the heap of earth in the centre of the "godown," but also in that in which the boiling pans and crystallising vats are set up, large amounts of saltpetre are "locked up."

*Nitrification in refinery earths.*—There is a generally accepted notion that saltpetre "grows" in the refinery earths, or put into more scientific language, that "nitrification" occurs in them.

This is a point on which we have no very definite evidence because the matter has never been really examined by the chemist and bacteriologist.

In the preceding paragraphs, it has been explained that considerable additions of nitrate are constantly made to the refinery earth and therefore such earth must necessarily contain large amounts of this salt. Hence, because nitrate is regularly extracted from this earth is in itself no proof that nitrification occurs in it. Indeed the amount of other salts present would rather suggest that no nitrification occurs. However this may be, the following data which I was able to obtain from two large refineries are of interest in showing how the "balance" of nitrate in a refinery stands.

During 4 months, refinery "B" bought 11,240 maunds of crude saltpetre containing about 55 per cent. of nitrate or say 6,180 maunds of pure potassium nitrate. From this 5,177 maunds of refined saltpetre were obtained, containing about 90 per cent. pure potassium nitrate equal to 4,660 maunds of the pure salt. Thus there was a deficit of some 1,520

maunds of nitrate. Secondly during the same period, 10,910 maunds of saltpetre were washed, which produced 9,772 maunds of the washed salt. The deficit is equal to about 10 per cent.; the sodium chloride was reduced by about 2 to 3 per cent. and it is certain that of the remaining 7 or 8 per cent., most was nitrate. Assuming 5 per cent. to be this salt, it is equal to some 500 maunds of nitrate. Thus there was a deficit of 1,500 *plus* 500 = 2,000 maunds of potassium nitrate on these two operations. During this period, also, the *refinery earth* produced only 1,716 maunds of saltpetre, containing some 60-70 per cent. of nitrate, or say (at 70 per cent.) 1,200 maunds of pure nitrate of potash. Thus, as a whole, some 2,000 maunds of nitrate disappeared in waste liquors, and 1,200 were recovered from the earth. Similarly at refinery "S" 6,674 maunds of crude saltpetre were refined, containing about 50-60 per cent. of potassium nitrate equal to (at 55 per cent.) 3,670 maunds of the pure salt. 3,267 maunds of refined saltpetre were obtained containing say 90 per cent. potassium nitrate equivalent to 2,940 maunds of the pure salt; or a deficit of some 730 maunds of nitrate occurred in the operation. At the same time, 6,065 maunds of refined saltpetre were washed, 5,060 maunds were recovered or a deficit of about 16 per cent. The sodium chloride decreased by some 6 per cent. and assuming only 5 per cent. of the remainder to have been nitrate, it is equal to 300 maunds of pure potassium nitrate. Together then some 1,030 maunds of pure nitrate disappeared in waste liquors. During the same period 1,360 maunds of saltpetre, of some 50 per cent. purity, was obtained from the earth, equal to about 680 maunds of pure nitrate. Thus taking the refinery figures as a guide there was a loss of nitrate and not a gain, during these operations. Admittedly these figures are not entirely reliable for the purpose, because the nitrate of the mother liquor which is thrown on the earth to-day is not extracted to-morrow, but probably remains for months before it is recovered. The refinery earth is only worked over gradually during the season. At the same time the figures quoted were those for several months' working and they point to a loss of nitrate, and do not lend any support to the idea of nitrification occurring in the earth. At the same time admitting that a loss of nitrate constantly occurs in a refinery, this is in itself no proof that nitrification does not take place. It is possible that the process is in existence, aiding in the supply of nitrate, but if so, then the total loss of nitrate in the refinery is even greater than available figures would indicate.

## PART III.

*Suggested Improved Method of Refining.*

The question naturally arises whether it is possible to suggest improvements in the methods of producing Indian saltpetre. Regarding the production of *crude saltpetre* it is practically impossible to suggest any useful alteration in existing methods. This is not because it is impossible to obtain from the Indian nitre earth a purer salt-petre than that produced by the Nunia, but because it is financially impossible. Better methods would require more expensive apparatus, far beyond the reach of these people, and would necessitate the transport of the earth to a central factory at such a distance that the cost of freight would more than counterbalance the other advantages gained. The Nunia works very economically and his process is the best that could be devised.

Turning to the *refining* of the *crude saltpetre* the circumstances are different. Most of these refineries deal with thousands of maunds (hundreds of tons) of *crude saltpetre* in the year, the value of which is considerable and renders it quite possible to employ good apparatus for more economical working. The chief defects in the present process are (i) the considerable amount of saltpetre which remains in the refinery earths, and which is only gradually recovered; this is obviously capital "locked up" and unremunerative for a long time. (ii) Owing to the comparatively weak liquors which are prepared, much water has to be evaporated and hence the consumption of fuel is large. (iii) The present processes are wasteful and cause a serious loss of nitrate. (iv) Although the "*kudmi*" saltpetre is of good quality, the "*kuthia*" saltpetre is of low quality. (v) The colour of the refined saltpetre is brown and it has to be washed in order to remove this colour. (vi) Refining is much hindered during the monsoon period.

Now it should be possible to remove these defects in a great measure. Supposing a suitable quantity of *crude saltpetre* is heated with mother liquor, the latter will dissolve, as has been explained, large quantities of potassium nitrate, but very little of the other salts. Supposing now the hot liquid is separated as completely as possible from the insoluble matters, and then cooled, the potassium nitrate will crystallise out. A small amount of impurities will crystallise out with it, at the same time, but their proportion will be small. The mother liquor can be then



# PLATE VIII.

## SALTPETRE REFINING APPARATUS

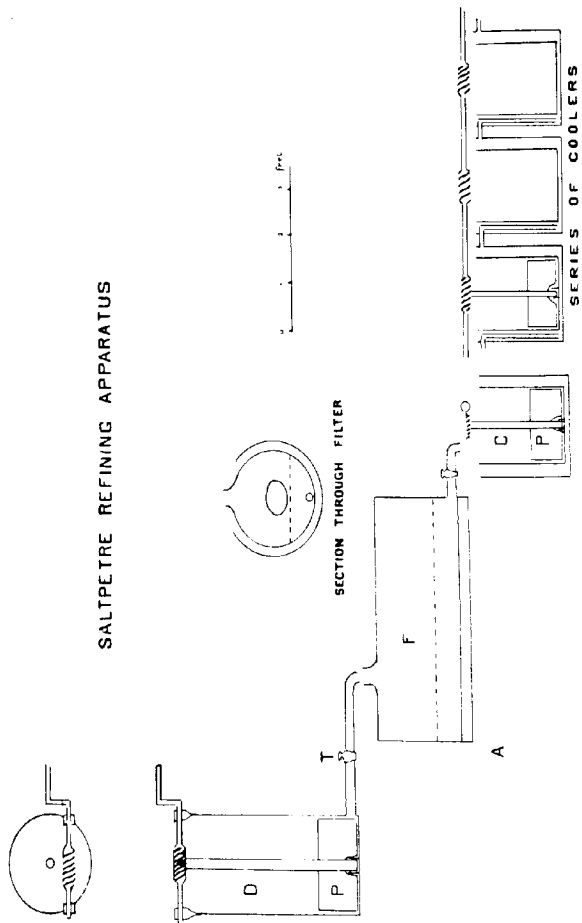


Fig. 10. Diagram of layout of the apparatus for the recovery of soda from saltpetre.

separated from the purified saltpetre and employed again to dissolve more nitrate from crude saltpetre. Thus it is possible, provided we have suitable apparatus, to separate the potassium nitrate from the crude saltpetre *without evaporating any water*. The liquors must be heated, but such heating requires far less fuel per pound of water than it does to *evaporate* one pound. For instance to heat each pound of water from 25°C to 100°C (the boiling point) requires only 75 heat units; but to evaporate the one pound of water requires 585 units; or we may say that to evaporate the one pound requires about seven times the amount of fuel that is needed to merely heat it to the boiling point.

Before employing a concrete example to illustrate the foregoing proposition, it will be best to describe the process which has been designed at the Agricultural Research Institute, Pusa. The operations are five in number. (i) The crude saltpetre is heated to nearly the boiling point with a suitable quantity of mother liquor in a special vessel, by which the whole of the potassium nitrate passes into solution, but the other salts remain almost entirely undissolved. (ii) This hot mixture of liquid and undissolved matters is run into a filter which is kept hot. The filter is then closed and air is pumped in. By thus increasing the pressure in the filter, the hot liquid is forced away from the undissolved matters. The latter will of course retain some part of the hot liquid, because it is never possible to separate the whole of a liquid from a solid in this manner. Hence some of the nitrate will also remain with these undissolved salts. (iii) The hot liquid from the filter is run into a vessel in which it is cooled quickly. By this means the refined saltpetre is precipitated as a mass of small crystals of very fair purity. (iv) The cooled mixture of saltpetre crystals and mother liquor is run into a centrifugal machine which separates the mother liquor almost completely, and the refined saltpetre comes out dry and white. (v) After the mother liquor has run out of the centrifugal, a small quantity of cold water is syringed on to the saltpetre (whilst the centrifugal is still running) and this removes most of the chlorides.

The diagram shows the apparatus in skeleton form (see plate VIII). In this D is the "Digester" in which the crude saltpetre is heated with mother liquor by the fire at A. In order to allow the liquid to heat quickly it is stirred by the paddle P. After it is hot the tap T is opened and the hot mass is run into the pressure filter F. This filter is a double vessel as indicated in the cross section. The inner vessel is the filter proper and has a

filter bed of wire gauze as indicated by the dotted line. The outer vessel contains water which is heated by the fire at A. It is essential that the filter shall be kept hot in order that the mixture of mud and liquor prepared in the digester shall not cool at all during filtration. After running in the hot mass from D, the mouth of the filter is closed and air is pumped in. (The pump is not shown in the diagram.) The hot filtered liquid runs from F into the cooler C. This cooler (of which there are several) is like the "digester" and has a paddle P, but it also stands in an outer vessel through which cold water is running, and by working the paddle the liquid is cooled quickly. When cold, the mixture of crystallised saltpetre and mother liquor is run out of the cooler C, into buckets, from which it is poured into the centrifugal, or it is run direct into the centrifugal. The latter is not shown in the diagram. This centrifugal then separates the mother liquor and retains the refined saltpetre. The latter is a perfectly white, fine grained crystalline powder, nearly dry, of about 90 per cent. purity. In order to still further purify it a small quantity of cold water, about 1 seer per 10 seers of saltpetre in the centrifugal, is syringed on to it and the purity of the product rises to 93 or 94 per cent. The insoluble "mud" left on the filter contains still a great deal of nitrate. It is removed from the filter and returned to the digester, where it is again heated with mother liquor and again passed into the filter. The amount of nitrate obtained from this second extraction is naturally less than from the first extraction. Some nitrate still remains in the mud, but it is now so little that it does not pay to extract it a third time in this apparatus.

In order to illustrate more completely the process, the following data may be quoted from the small model refinery which has been working at the Allahabad Exhibition.

(i) 7.75 maunds of crude saltpetre, containing 30 per cent. of potassium nitrate, yielded 2.6 maunds of refined saltpetre of 90 per cent. purity.

(ii) 17.4 maunds of crude saltpetre, containing 45 per cent. of potassium nitrate, yielded 6.5 maunds of refined saltpetre of 90 per cent. purity.

(iii) 5.5 maunds of crude saltpetre, containing 53 per cent. of potassium nitrate, yielded 2.3 maunds of refined saltpetre of 90 per cent. purity.

(iv) 6.8 maunds of crude saltpetre of 46 per cent. purity yielded 2.4 maunds of refined saltpetre of 93 per cent. purity. The maund here referred to is the standard maund of 40 seers.

*Cost of the apparatus.*—A complete apparatus for refining about 30 maunds of crude saltpetre per day will cost about **Rs. 3,000** and would best be obtained from Messrs. Arthur Butler and Co., Mozufferpore, who made the small apparatus which has been working at the Exhibition. This is not a large capital expenditure. If depreciation be put at 25 per cent. the annual charge is **Rs. 750**. If work continues for 300 days per annum, 9,000 maunds would be dealt with, or the cost of apparatus per maund of crude saltpetre is equal to about annas  $1\frac{1}{2}$ .

*The mud.*—The process as described leaves the mud to be dealt with. This contains about 15 to 20 per cent. of potassium nitrate and may be extracted in the "*Kurias*" as at present. But it would not be difficult to improve this part of the process, if the apparatus which has been described were adopted in any refinery.



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